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1 **Electroconductivity and structural-phase changes of anodic alumin implanted by titanium and molybdenum**
Abramov, I.I.; Danilyuk, A.L.; Litvinovich, G.V.; Sokol, G.V.; Burova, E.A.; L V.V.; Cherenda, N.N.
Microwave Conference, 1999. Microwave & Telecommunication Technology. International Crimean [In Russian with English abstracts] , 1999
Page(s): 289 -291

[Abstract] [PDF Full-Text (580 KB)] **CNF**

2 **Low contact resistance of 30 nm and 200 nm diameter Bi wire arra composites**
Huber, T.E.; Graf, M.J.; Foss, C.A., Jr.
Thermoelectrics, 1999. Eighteenth International Conference on , 1999
Page(s): 558 -561

[Abstract] [PDF Full-Text (356 KB)] **CNF**

3 **Thermoelectric bismuth wire array composites**
Huber, T.E.; Calcao, R.
Thermoelectrics, 1998. Proceedings ICT 98. XVII International Conference o
Page(s): 244 -246

[Abstract] [PDF Full-Text (256 KB)] **CNF**

4 **Development of a cost effective high performance metal QFP packa system**
Mahulikar, D.; Pasqualoni, A.; Crane, J.; Braden, J.

Low Contact Resistance of 30 nm and 200 nm Diameter Bi Wire Array Composites

T.E. Huber[1], M.J. Graf[2], and C.A. Foss, Jr[3]
[1] Department of Chemistry, Howard University, 500 College St. N.W., Washington, D.C. USA*,
[2] Department of Physics, Boston College, Chestnut Hill, MA, USA,
[3] Department of Chemistry, Georgetown University, Washington, D.C. USA,

We have prepared Bi wire arrays with diameters between 200 nm and 30 nm in a matrix of porous anodic alumina. The wire array conductivity down to 0.3 K has been measured as well as its magnetoresistance up to 8 T. Our measurements indicate that the wires are good conductors in the temperature range investigated.

Introduction

There is recent interest in the thermoelectric properties of Bi nanostructures. It has been reported that bulk Bi, a semimetal, has the largest thermomagnetic figure of merit $z_E T$ at moderately low temperatures (~100 K) and magnetic field of 1 T. [1] Alloying Bi with Sb has the effect of transforming the material into a shallow semiconductor that exhibits the largest known thermoelectric figure of merit zT at low temperatures. Nanostructured phases of these materials may perform even better than their bulk counterparts because the phonon thermal conductivity is reduced at grain boundaries and interfaces. [2] Also, the figure of merit of low dimensional systems may be increased over that for bulk because of the increase of the electronic density of states. [3] The physics involved results mainly because Bi has the smallest electron effective mass among all known materials and therefore quantum confinement in Bi are more manifest in nanowires of larger diameter than those of any other nanowire material. It is known that electron confinement in a thin film of thickness t~30 nm promotes a semimetal-to-semiconductor (SMSC) transition in pure Bi. The SMSC has been discussed in connection with Bi nanowires, [4] although never proven experimentally in a conclusive manner. Clearly, confinement introduces electronic scattering and the mode of electron transport, i.e. localized or ballistic, is relevant for thermoelectricity because the predicted increase in zT occurs only if mobility is not adversely affected by the confinement. In the case of carbon nanotubes transport studies on samples with low-ohmic contacts show that nanotubes are capable of displaying little resistance and can sustain high current densities (~10^9 A/cm^2). [5]

In this work Bi quantum-wire arrays were fabricated by the pressure injection technique, which can produce continuous, dense nanowire arrays, as required by thermoelectric applications. Like many other conductors, molten Bi does not wet insulators such as alumina or silica. Therefore, molten Bi will not spontaneously penetrate the channels of an insulating matrix. We have developed a technique that utilizes nanostructured insulators as a matrix for the synthesis of dense composites using high-pressure injection of the semiconductor melt. Two types of anodic alumina templates have been used in this work. One type is a commercial membrane sold for microfiltration under the trade name Anopore [6]. It consists of an alumina plate 25 mm in diameter and about 55 μm thick, which support an array of parallel, largely non-interconnected, cylindrical channels running perpendicular to the plate surface. The second type of porous anodic alumina used in this work was prepared in our laboratories by anodizing a high-purity aluminum substrate in acid solutions.

The Laplace equation [7] gives the excess pressure needed to inject channels of diameter d: $P = 4\gamma/d$. Here γ is the surface tension. Taking the surface tension of 380 dyn/cm [8], the equation above becomes $d = 12/P$ where P is measured in kilobars and d is in nanometers. Thus, for a modest applied pressure of 1 kbar, for example, all channels larger than 12 nm in diameter will fill with liquid Bi. The design and operation of the injection apparatus have been described elsewhere. [9] The setup's pressure is 20,000 PSI.

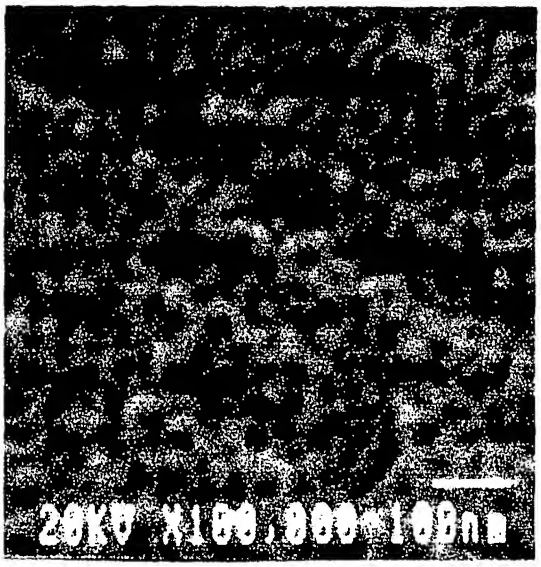

Fig. 1. Top view of the 30 nm Bi diameter wire array. Dark areas represent alumina.

A scanning electron microscope (SEM) image of the 30 Bi diameter wire array is shown in Fig. 1. Our image is not the best quality because of the required magnification factor of 100,000. The crystalline direction of the Bi wires is relevant to their

thermoelectric applications, since the thermal and electrical conductivities of Bi, as well as the Seebeck coefficient are anisotropic. For example the thermal conductivity λ at 100 K is 18 $Wm^{-1}K^{-1}$ along the trigonal direction and 13 $Wm^{-1}K^{-1}$ normal to the trigonal direction. [1] Our X-ray diffraction spectra show strong scattering peaks for 22.48°, 45.88° and 71.50° corresponding to the lattice planes with the Miller indices (003), (006) and (009), respectively. The enhancement of the intensity of these peaks can be contrasted with the scattering peak with the Miller index (102), which is the strongest for bulk Bi and has medium intensity in our nanowire spectra. This indicates that the nanowires crystal grains are oriented with the trigonal axis along the wire length. Zhang, Sun, Dresselhaus, and Ying [4] studied ultrafine Bi nanowire arrays with wire diameter of 65 and 110 nm. They studied the orientation of the nanowires with X-rays using the same geometry that we have used. They find that their highest intensity peak is the (202) peak at 48.70° with very weak scattering for the angles corresponding to the (003), (006) and (009) indices. In contrast, the (202) peak in our spectra is weak.

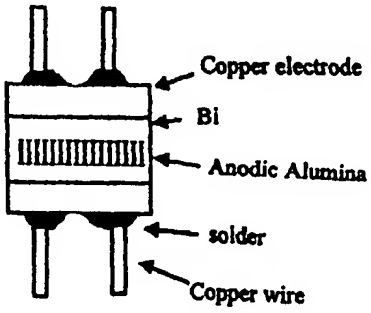

Fig. 2. Four contact devices used in this work.

With a view at thermoelectric applications, we looked for a way to minimize the electrical resistance of contacts to the wires. Since bulk Bi is a good conductor we took advantage of the injection process to create a Bi layer between the metal electrodes and the wire array. The method that we have employed is illustrated in Fig. 2. Electrical contact was made via copper leads attached to copper electrodes. The space between the electrodes and the template, as well as the template, was injected with molten Bi. This resulted in a thin (10 μm) layer of Bi between the copper electrodes and the wire array. We have presented our 200-nm diameter wire array magnetoresistance data previously. [10]. The sample resistance was measured by injecting an a.c. current (100 Hz) using one pair of contacts in opposite sides of the sample. The voltage drop is detected on another pair of opposite contacts on the copper electrodes. In this manner the lead resistances do not affect our measurements. The Bi wires-copper contact resistance is estimated to be a few μΩ. At 300 K the resistance of the 200-nm wire arrays sample is roughly 65 μΩ. Since the wire array area is 0.03 cm^2 and the composite thickness is 55 μm, the composite resistivity is $3.5x10^{-4}$Ω-cm. Since the Bi volume fraction is 0.5, the room temperature resistivity of Bi in the channels is $1.8x10^{-4}$Ω-cm. The electrical resistivity of bulk Bi along the trigonal direction is $1.1x10^{-4}$Ω-cm [1]. Therefore the ratio of the room temperature resistivity of the Bi nanowire to that of the bulk is 1.6. Our 200-nm wire diameter sample has higher conductivity than similar samples reported in the literature. For example, Liu et al [11] report a fivefold to tenfold increase in the room temperature resistivity of their 400 nm nanowires compared to that of bulk Bi. The resistance ratio R(300 K)/R(4 K) is the results of competing effects: the carrier density increases and the mobility decreases with increasing temperature [10]. Our 200 nm samples exhibit a R(300 K)/R(4 K) that is roughly 2, indicating reduced scattering. This observation is in approximate agreement with Gurvitch measurements for 200-nm diameter single wires [12]. Therefore we are confident that we have found a way to fabricate samples that minimize the contact resistance of the Bi nanowires using the technique of sandwiching the nanowire arrays. In order to evaluate better this technique we have also prepared samples of Bi in 200-nm Anopore without copper electrodes. We find that the magnetoresistance of the bulk Bi at both sides of the nanowire array is significant in comparison with the nanowire array resistance. The 30-nm diameter wire array resistivity is shown in Fig. 3. Taking into account that the

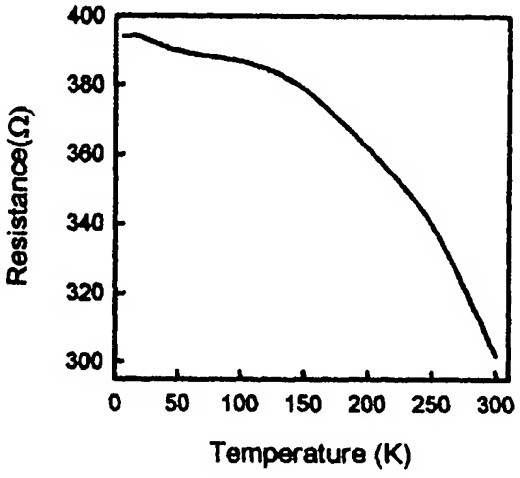

Fig. 3. 30-nm Bi wire array resistance as a function of temperature

sample cross-sectional area is 0.02 cm^2 , the wire length is 40 μm, and the filling fraction is 50%, the resistance of the room temperature resistivity of the Bi nanowire to that of the bulk is 7.

The temperature-dependent resistivity exhibits a negative temperature coefficient. In comparison, the bulk Bi exhibits obeys a T^2 law at low temperatures and is roughly proportional to T for T>100 K. [1] Therefore the behaviour we observe is unlike that of the bulk Bi. The negative temperature coefficient of the temperature dependent resistance is reminiscent of the effects one expects for a semiconductor. However, the observed R(T) clearly fails to display the thermal activated behaviour typical of a semiconductor. In fact, one can understand the temperature dependent coefficient by analogy with thin films, [13] in terms of a classical finite-size effect caused by the limitation of the carriers mean free path by scattering and the wire surface and grain boundaries.This is expected to be strong since the wire dimensions are much less than the bulk mean free path. To further complicate the interpretation of the magnetoresistance data there is the matter of the doping of the nanowires due to surface charges, a well known factor in thin films. [13] These effects could very well mask the SMSC transition in Bi nanowires. Clearly, a careful study of these matters is beyond the scope of this paper and will be studied in a future publication.

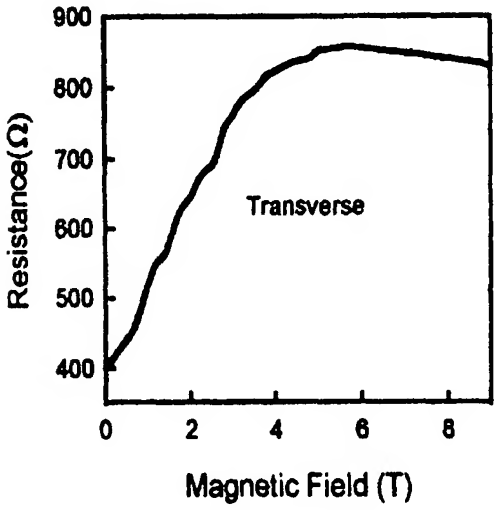

Fig. 4. The magnetic field dependence of the resistance of a 30 nm diameter wire array for T= 6 K.

The plateau of the resistance at 120 K is very interesting. In comparison, the 200-nm samples show a plateau at 40 K. If one accepts the explanation that the plateau at 40 K [13] arises from the quantum size effect (QSE), then the increase in the plateau temperature for smaller diameter wires may be attributed to the increased energy gaps between the various energy subbands for the smaller diameter wire. However, we are not aware of careful theoretical calculation of these effects for a realistic band structure. We have also studied the effect of a longitudinal and transverse magnetic field on the resistance, up to a field of 9 T. It should be noted that the magnetoresistance MR=R(T,H) –R(T,0), where R(T,H) is the resistance at a given temperature and magnetic field, is positive. The transverse MR is different from that of bulk Bi that exhibits a $B^{1.6}$ dependence in the high magnetic field range [14] whereas our MR is saturating. We have observed that the 30-nm Bi wires exhibit a reduced MR in comparison with the 200-nm Bi wire MR. Clearly the details of these measurements are outside the scope of the present paper and will be presented elsewhere.

Conclusions

To summarize, by pressure-injecting molten Bi into the nanochannels of an anodic alumina template we have successfully fabricated Bi nanowire arrays with controlled wire diameters (32 nm and 200 nm) and high packing densities of roughly 50%. Nanowires in the array are crystalline and show a high degree of orientation.These structural characteristics, when combined with the small effective mass of Bi and its high thermal conductivity relative to that of the alumina matrix, make the Bi nanowires arrays especially promising for thermoelectric applications. Low contact resistance configurations, with contact resistance less than 10 μΩ, have been prepared. The resistivity, which is non-metallic (dR/dT<0), shows a plateau at 120 K which is interpreted in terms of size quantization. We did not find evidence for the SMSC transition. Clearly, smaller diameter wires are highly desirable as thermoelectric materials and it appears that it is possible to extend our studies to alumina-based wire arrays of even smaller diameter than 30 nm.

Anknowledgements

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Low contact resistance of 30 nm and 200 nm diameter Bi wire array composit

- Huber, T.E. Graf, M.J. Foss, C.A., Jr.
Dept. of Chem., Howard Univ., Washington, DC, USA

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On page(s): 558 - 561
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Baltimore, MD, USA
1999
ISBN: 0-7803-5451-6
IEEE Catalog Number: 99TH8407
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INSPEC Accession Number: 6663342

Abstract:
We have prepared Bi wire arrays with diameters between 200 nm and 30 nm of porous anodic alumina. The wire array conductivity down to 0.3 K has beer as well as its magnetoresistance up to 8 T. Our measurements indicate that tl are good conductors in the temperature range investigated.

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Electronic Components and Technology Conference, 1993. Proceedings., 43r
Page(s): 405 -411

[Abstract] [PDF Full-Text (424 KB)] **CNF**

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5 **A new a-Si TFT with Al/sub 2/O/sub 3//SiN double-layered gate i for 10.4-inch diagonal multicolor display**
Yamamoto, H.; Matsumaru, H.; Shirahashi, K.; Nakatani, M.; Sasano, A.; Kc Tsutsui, K.; Tsukada, T.
Electron Devices Meeting, 1990. Technical Digest., International , 1990
Page(s): 851 -854

[Abstract] [PDF Full-Text (248 KB)] **CNF**

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6 **Improved coating for amorphous alloy with low loss deterioration**
Okazaki, Y.; Kanno, H.; Sakuma, E.
Magnetics, IEEE Transactions on , Volume: 25 Issue: 5 , Sept. 1989
Page(s): 3352 -3354

[Abstract] [PDF Full-Text (248 KB)] **JNL**

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7 **Multilevel metallization for large area point-contact solar cells**
Verlinden, P.; Swanson, R.M.; Sinton, R.A.; Kane, D.E.
Photovoltaic Specialists Conference, 1988., Conference Record of the Twenti 1988
Page(s): 532 -537 vol.1

[Abstract] [PDF Full-Text (452 KB)] **CNF**

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8 **Perpendicular magnetic flexible disc by anodic oxidation**
Tsuya, N.; Tokushima, T.; Shiraki, M.; Umehara, Y.; Saito, Y.; Nakamura, H Y.
Magnetics, IEEE Transactions on , Volume: 24 Issue: 2 Part: 2 , March 1988
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L16	L14 and puls$4	298	L16
L15	L14 puls$4	906413	L15
L14	L13 and (volt$4 or current$1 or power)	828	L14
L13	L12 and (desor$6 and adsor$6)	1047	L13
L12	(sensor or detector) and (microheater or heater or (resistive near4 heat$3))	79665	L12
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L6	L5 and (AAO or (anodic adj alumina) or (anodic adj aluminum adj oxide))	0	L6
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L2	((73/1.02 |73/1.06 |73/23.2 |73/29.01 |73/29.02 |73/335.02 |73/335.03 |73/335.05 |73/31.05)!.CCLS.)	2020	L2
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L15	L14 puls$4	906413	L15
L14	L13 and (volt$4 or current$1 or power)	828	L14
L13	L12 and (desor$6 and adsor$6)	1047	L13
L12	(sensor or detector) and (microheater or heater or (resistive near4 heat$3))	79665	L12
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L8	(AAO or (anodic adj alumina) or (anodic adj aluminum adj oxide))	421	L8
DB=USPT; PLUR=YES; OP=OR			
L7	L5 and anodic$4	103	L7
L6	L5 and (AAO or (anodic adj alumina) or (anodic adj aluminum adj oxide))	0	L6
L5	L2 or L3 or L4	5067	L5
L4	((204/424 |204/425 |204/426 |204/431)!.CCLS.)	1275	L4
L3	((29/592.1 |29/593 |29/825)!.CCLS.)	1868	L3
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- Govyadinov, A.N. Zakhvitcevich, S.A.
Vacuum Microelectronics Laboratory, Institute of Electronics Belarus Academy Sciences

This paper appears in: Vacuum Microelectronics Conference, 1997. Technical I 1997 10th International
On page(s): 735 - 738
August 17-21, 1997
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26 **Field Emitter Arrays Based On Natural Selforganized Porous Anodi Alumina**
Govyadinov, A.N.; Zakhvitcevich, S.A.
Vacuum Microelectronics Conference, 1997. Technical Digest., 1997 10th Int

Page(s): 735 -738

[Abstract] [PDF Full-Text (464 KB)] **CNF**

27 **Anodic Alumina: A Promising Constructional Material For Vacuum Semiconductor Microelectronics**
Mukhurov, N.I.; Efremov, G.I.; Kotova, I.F.
Vacuum Microelectronics Conference, 1997. Technical Digest., 1997 10th Int

Page(s): 617 -621

[Abstract] [PDF Full-Text (728 KB)] **CNF**

28 **Development of aluminum electrolytic capacitors for EV inverter applications**
Efford, T.; Shibata, Y.; Kanezaki, A.
Industry Applications Conference, 1997. Thirty-Second IAS Annual Meeting, Conference Record of the 1997 IEEE , Volume: 2 , 1997
Page(s): 1035 -1041 vol.2

[Abstract] [PDF Full-Text (472 KB)] **CNF**

29 **Computer-aided design of asymmetric stripline for multichip modı**

ANODIC ALUMINA: A PROMISING CONSTRUCTIONAL MATERIAL FOR VACUUM AND SEMICONDUCTOR MICROELECTRONICS

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In respect of its basic electrophysical characteristics, anodic alumina is close to leuco sapphire and glass ceramics widely used in microelectronics. However, there are other inherent properties which are unique for this material. Anodic alumina substrates are used to produce multilevel large-format circuit boards for custom hybrid semiconductor microcircuits, thermionic integrated circuits, microcommutation electrostatic devices, etc. Porous anodic alumina substrates hold promise for formation of multi-point field emitter arrays where each cell has dimensions of several thousand angstrom/ This opens up the possibility of making high-quality displays and super-miniature microwave devices and integrated microcircuits which outperform the existing devices.

INTRODUCTION

In its electrophysical properties, alumina produced by anodic oxidation of aluminium is very close leuco sapphire and glass ceramics widely used in microelectronics. Additionally, it has a range of other unique properties [1]. Along with the possibility of making high-quality dielectric layers in a wide thickness range 10^{-2} - 10^{3} μm, the aluminium anodizing technology in conjunction with photolithography, etching and vacuum deposition processes makes it possible to form holes, cavities and many other elements within dielectric volume on different levels and with high degree of accuracy. They are used as support and insulating power elements of electron device structures, and are employed to form gaps, grroves and through windows of various shapes. Also based on anodic alumina (AA) substrates are multilevel large-format circuit boards used to produce custom hybrid semiconductor microcircuits for various applications, including the microwave range [2]. Thermionic vacuum integrated circuits (VIC) made so far are structurally based on volumetric microrelief anodic alumina substrates on which film microtriodes, passive elements and interconnections are formed [3]. The possibility of making both logic and analog VIC's and expanding their functional capabilities by adding microcommutation electrostatic devices and static electricity sensors based on them has been demonstrated in [4].

The use of porous AA substrates is particularly promising for formation of multi-point field emitter arrays in which the size of each cell lies within several thousand angstrom. This opens up good prospects for modelling and fabrication of high-quality displays and super-miniature microwave devices and intergrated microcircuits with highly advanced parameters.

1. SPECIFIC PROPERTIES OF ANODIC ALUMINA

Anodic alumina is characterized by sufficiently high mechanical properties. Numerically, they are substantially dependent on chemical and thermal conditions of oxide substrate formation. In what follows, all characteristics and parameters are given for AA substrates in an uncombined state. i.e. detached from aluminium after electrochemical oxidation down to the required thickness. The source AA material is X-ray amorphous [1]. Following thermal treatment at temperatures higher than 820 °C it acquires a polycrystalline state featuring a number of γ-, δ-, θ- и α-high-temperature modifications. The first three modifications are very similar in their characteristics and are all encountered in the AA. α-oxide is more like Al_2O_3-based ceramics, and, because of its porosity, is less stable in extreme environment as compared to other AA modifications. Therefore, characteristics of the latter material will not be considered hereinafter.

Averaged data for alumina prepared in 3% aqueous solution of oxalic acid. Our investigations have shown that in such an electrolyte γ-AA substrates are formed with mechanical

strength and stability in extreme environment that are suitable for subsequent fabrication of VIC's [3].

At room temperature, bending strength σ_b of the source X-ray amorphous AA is 380 MPa, gradually increasing to 400 MPa with temperature rising to 600 °C. For γ-oxide, the same parameter changes from 370 MPa to 300 MPa, which is somewhat lower than its value typical of amorphous specimens. Hardness is another essential characteristic of materials. Microhardness of amorphous oxide is strongly dependent on anodizing conditions. As anodizing temperature changes from 10 °C to 30 °C (electrolyte concentration is 3%), the microhardness H_v falls down from 4700 MPa to 2100 MPa. As acid concentration in electrolyte is increased (solution temperature is 10 °C) from 3% to 5%, microhardness rises from 4800 MPa to 5800 MPa. However, a further rise of the concentration to 8% it falls down to 4400 MPa. These dependences can be used to obtain substrates with required hardness and to determine technological parameters tolerances. Microhardness is also substantially affected by thermal treatment. Thus, γ-oxide has microhardness in the range of 5000-6000 МПа. The increased microhardnes value results in a lower bending strength of the γ-oxide due to enhanced influence of surface microdefects which are responsible for stress concentration. Oxide hardness does not depend on the hardness of the source aluminium, which changed from 370 to 290 MPa at thermal straightening temperature in the range of 100-200 °C.

The modulus of elasticity of amorphous oxide and γ-oxide, as determined from the experimental of sag-vs-load dependence, are practically uniform in value and equal to 140 GPa. Fig. 1 presents temperature dependence of thermal expansion coefficient β of γ-oxide, which demonstrates a linear behaviour. Like ceramics, AA demonstrates dimensional changes, so-called shrinkage, when it is subjected to thermal treatment and suffers transformation from amorphous to polycrystalline state. Depending on particular design and substrate configuration, shrinkage can be as high as ±0,4%.

The following Table illustrates basic electrophysical properties of anodic alumina as a dielectric material.

AA type	ρ_v, Ohm·m	ρ_s, Ohm/□	tg δ (f=10^3Hz)	ε (f=10^3Hz)	$E_{пр.}$, MV/m
Amorphous	$5 \cdot 10^{10}$	10^{15}	$5 \cdot 10^{-3}$	6,2	45-75
γ-oxide	10^6-10^7	10^{12}	$100 \cdot 10^{-3}$	6,4	48-60

These data were obtained by using standard measurement methods, values of ρ_v, ρ_s, tg δ having been measured at d.c. voltage of 100V and frequency of 10^3 Hz. Relative dielectric permittivity was calculated from measured capacitance values. Higher values of the electric strength $E_{пр.}$ were obtained in special anodizing regimes. Fig. 2 shows frequency dependence's of ε' и ε", illustrating that AA values are close to ceramics values, thus, justifying the use of AA as a quality dielectric material in the microwave range .

2. EXAMPLES OF USING ANODIC ALUMINA AS PASSIVE AND/OR ACTIVE DIELECTRIC IN MICROELECTRONIC DEVICES

Multilevel AA substrates on aluminium base are employed in making hybrid integrated circuits (HIC) for microwave applications which impose stringent requirements on their electrical, thermal and mass-size characteristics [2]. A typical example of a HIC made on anodized aluminium substrate which also serves as a base of vacuum package is given in Fig. 3. Such substrates with holes to fit power transistors are used for making custom HIC's.

Of special interest is the use of AA substrates for formation of high-precision dielectric bases of light-emitting arrays and high-power semiconductor emitters. Advantages offered by such substrates as compared to conventional glass ceramic wafers are quite obvious from higher thermal expansion coefficient of AA and universal nature of its technology. The latter offers a

relatively simple formation of precision dielectric components with multi-level commutation which is rather attractive for light-emitting diodes. Its another advantage is associated with the possibility of combining the merits of semiconductor and vacuum microdevices.

Fabrication of efficient thermionic VIC's capable of operating in extreme environment (temperature up to 600 °C, radiation levels up to 10^9 R, 10^{16} N/cm^2 and above, pressure - from 10^{-6} mm Hg to 100 atm, high mechanical loads, etc., including simultaneous effects of all these impacts) could only be achieved through the use of AA as an active structural material. All previous attempts at making VIC's based on conventional ceramic materials or HIC's based on wide bandgap semiconductors or miniature ceramic microtriodes were not successful, prototypes being unable to survive operation in the conditions of severe environment [5]. The basic cause of failure of such integrated circuits was degradation of contacts of active and passive elements. With AA-based thermionic VIC's, active and passive elements are formed in one uniform technological cycle based on films of high-temperature conductive, dielectric and resistive materials, which are, where possible, compatible in their thermal expansion coefficients. Therefore, the resulting VIC's are potentially more stable to the effects of destabilizing factors.

Some examples of logic and analog thermionic VIC's based on AA are demonstrated in Fig. 4(a-d). A self-excited high-frequency VIC oscillator in metalloceramic package (Fig. 4 a) demonstrates operation at testing temperatures of 500 °C for as long as several thousand hours. A similar VIC of a multivibrator was able to work at radiation levels of 10^9 R, 10^{15} N/cm^2. Design principles and fabrication technology of the more intricate circuit components, e.g. a.c. amplifier and operational amplifier, are practically identical to those of the tested VIC's. Therefore, one can be sure that they will also survive operation in extreme environment.

The use of porous AA matrix for making multi-point field emitters seems to be another promising idea. The example of a field-emitter array is illustrated in Fig. 5. Such arrays could be used as prototypes for modelling miniature active components of VIC's, primarily for microwave range, with possible fabrication of high-quality displays in their basis.

CONCLUSION

A unique aluminium oxide technology and its product - anodic alumina substrates both with and without aluminium base - hold much promise for semiconductor and vacuum microelectronics applications and, undoubtedly, will have brilliant future.

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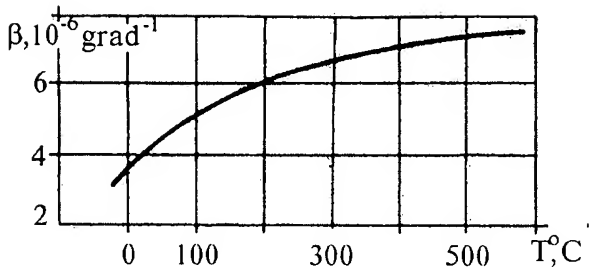

Fig. 1. Thermal expansion coefficent of γ-oxide- versus-temperature

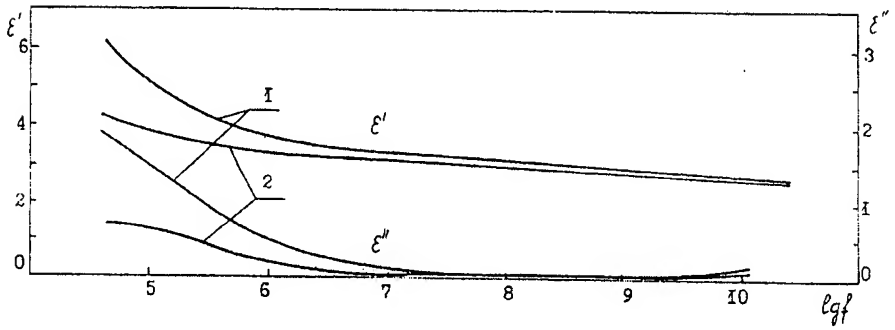

Fig. 2. Frequency dependences of ε' и ε" for amorphous alumina and и γ-oxide

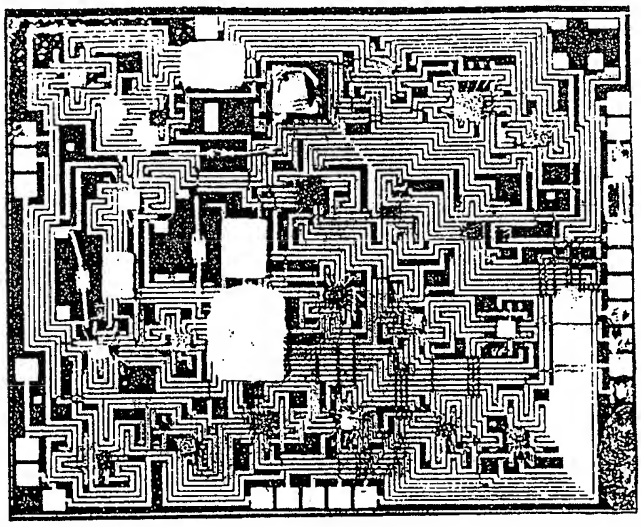

Fig. 3. Typical example of an HIC based on anodized aluminium substrate

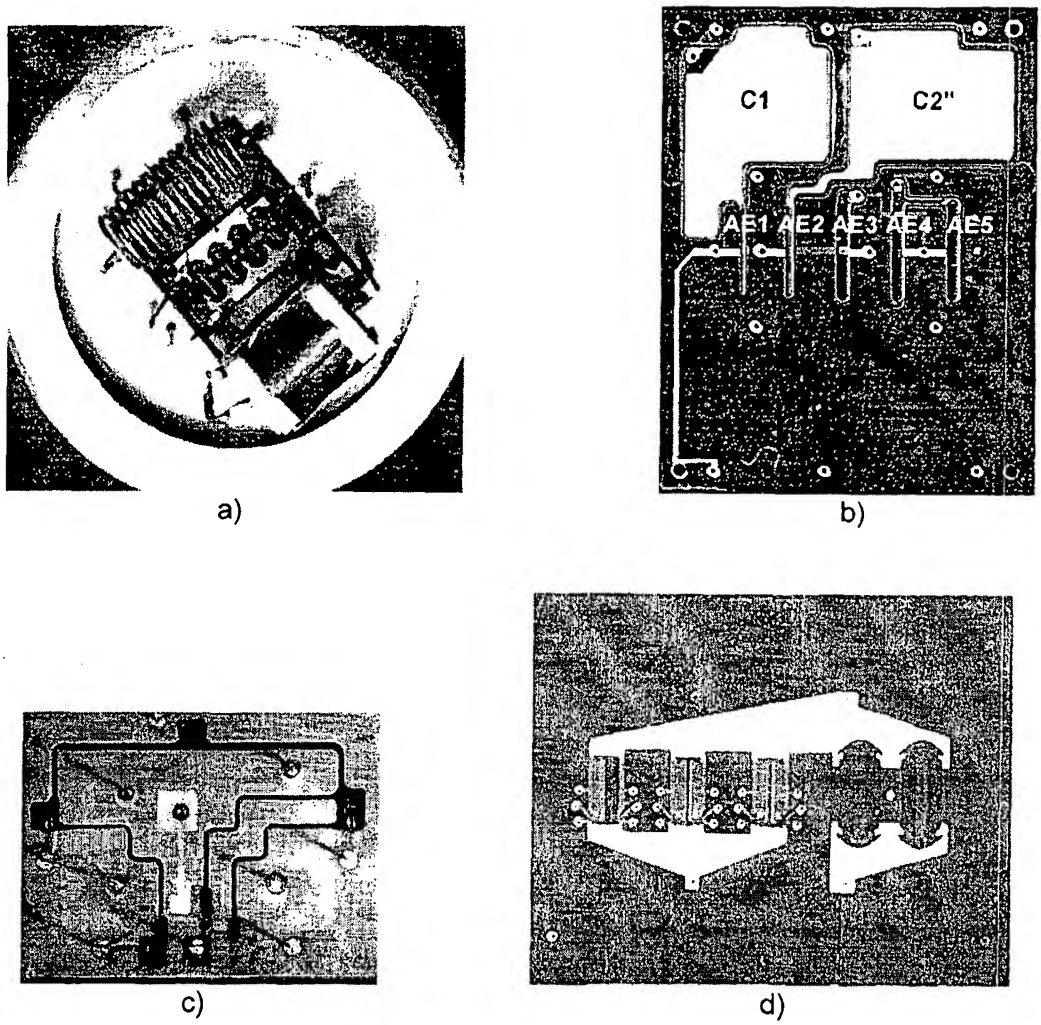

Fig. 4 (a-d). Examples of logic and analog thermionic VIC's made on AA substrates: a) self-excited high-frequency oscillator in metalloceramic package, b) a.c. amplifier with five amplifying stages, c) "2OR-NOT" logic circuit with three stages, d) a simple operational amplifier with three differential stages based on 3D active deflectron-type elements and two final stages based on 3D active elements

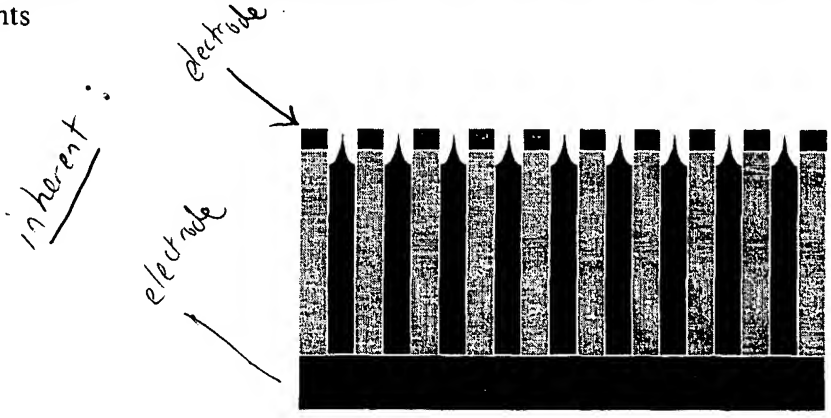

Fig. 5. Example of an AA-substrate-based field-emitter array.

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FIELD EMITTER ARRAYS BASED ON NATURAL SELFORGANIZED POROUS ANODIC ALUMINA

From: Evan Pert 703 306 5699

Alexander N. Govyadinov, Sergej A. Zakhvitcevich
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The FEA conception with tip's density up to 10^{10} cm^{-2} based on natural porous anodic alumina is proposed and its realizability is demonstrated. The basic technological steps of FEA based on the anodic alumina is described.

FYI: see "one electrode 5" at next page

1. INTRODUCTION

The main developments on creation of FEAs were carried out on the basis of Spindt's and silicon tecnologies [1,2]. All of them use artificial and expensive methods of microtechnology such as submicron lithography, selective etching (reactive ion etching) etc. Elements packaging up to 10^8 per sq. cm is achieved.

The further reduction of working elements (up to nanosize) in vacuum electronic devices, determining by requirements of nowadays, demands development of new technology. One of the interesting directions in this field is creation of nanodimernsional devices on the basis of self-organizing structure of polysilicon and anodic alumina films [3,4].

The goal of this study is to show a suitability of natural nanotechnology of formation a FEAs on the base of anodic alumina suggested in [5].

2. ANODIC ALUMINA STRUCTURE

The anodic alumina (AA) is widely used for a long time in various areas of electronic engineering. It is caused first of all by its good mechanical and electro-physical properties. Nevertheless use of its unique regular structure is limited by production of membrane-sieves and substrate for microfabrication in vacuum microelectronics [6-7].

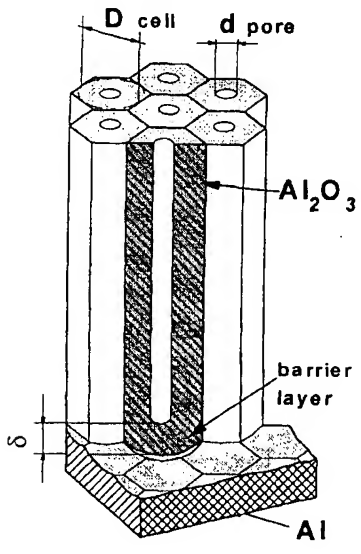

Fig. 1. Idealised model of porous anodic alumina structure.

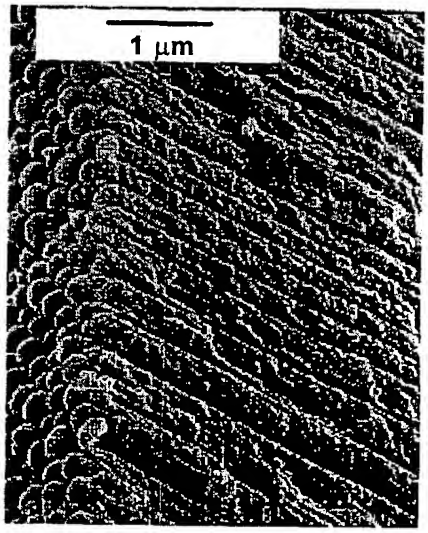

Fig.2. Micro graph of the anodic alumina film cross section.

Growth of an oxide during electro-chemical oxidizing results in formation regular cellular-porous insulating film. The film consists of a compact barrier layer directly attaching to metal and located over it thick porous layer, made of honeycomb cells (fig.1). The separately taken cell represents honeycomb with a central axial aperture, reaching up to barrier layer. The real structure of a porous anodic alumina film is presented in fig.2. It is very closed to idealised model.

The size of honeycomb oxide cells is determined first of all by a forming voltage and can be varied in an interval from 30 up to 700 nm, the diameter of aperture in a cell

from 10 up to 300 nm [8]. The thickness of anodic alumina films can be 0.1-100 μm. Hence, the process of the porous anodic alumina films producing is very simple and controllable and the natural cellular structure of porous anodic alumina well approaches as a natural mould for creation of unique FEAs with nanosized tips.

3. FABRICATION

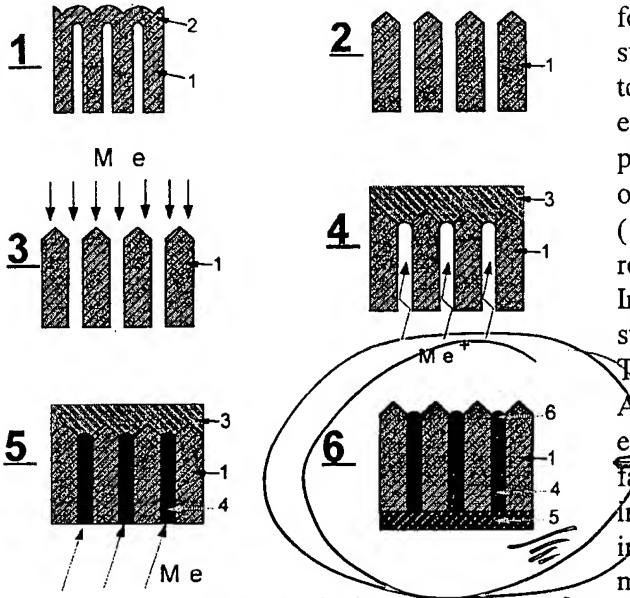

Fig. 3. Technological steps of nanodimensional FEA fabrication: 1- AA separation from Al support; 2- pores "opening"; 3- vacuum deposition of intermediate layer; 4- pores filling; 5 - base electrode forming; 6 - intermediate layer

The idea of nanodimensional FEA fabrication is following: use of natural anodic alumina porous structure with the removed barrier layer as a mould to incorporate a metal viscers in pores to form emission tips. The brief technological diagram of the proposed process is given in a fig. 3. Large-cellular oxide was used as a mould. After separation of oxide (1) from aluminium substrate a barrier layer (2) was removed to create through cylindrical pores. Intermediate layer of metal (3) was deposited onto a surface after pores 'opening' by sputtering method. The metal viscers (4) were incorporated into pores of AA matrix by electrochemical deposition. A base electrode (5) for electrical connection of tips was fabricated by vacuum deposition as well as intermediate layer. At a finishing stage the intermediate layer of metal was lifted up and the metal tips (5) were formed. The size of tips (their diameter) was determinated by pores of AA and the processing of barrier layer under its 'opening'.

4. RESULTS AND DISCUSSION

The size of the anodic alumina cells is determined by a forming voltage [8]. Thus, it is necessary for the process of anodic oxidizing of aluminium to carry out at high forming voltage to produce the largely cellular anodic alumina. AA film with the thickness from 10 up to 60 microns were formed in galvanostatic mode at a final voltage ~200 V. The mean size of cells was ~550 nm at density ~$4 \cdot 10^8$ cm^{-2}.

Fig.4. Cross section of typical porous AA for FEA fabrication.

The removal of a barrier layer and formation of the AA mould is the key moment for the FEA creation. We developed an electrochemical method of the barrier layer eliminating [9], which has appeared to be very fruitful. Its advantage is the simplicity and very high speed of process and, as a consequence, practically complete absence of changes in AA porous structure. The typical photo of AA cross section after 'opening' of barrier layer is presented in fig. 4.

As it was shown earlier [4], size and uniformity of FEA' tips based on AA technology were far from necessary. A problem was in large crystals of thin intermediate layer deposited onto a porous surface of the oxide mould. The size of grains determinates the tips' shaping. The film with the smallest grains is appreciated for FEAs preparation with uniform tips. For this purpose it is necessary to carry out the deposition of refractory metals at low

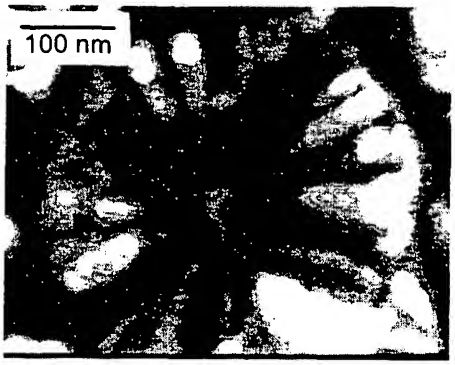

Fig.5. Top view of a single tip in typical FEA realised on the base of anodic alumina

temperature of the AA substrate to reduce migration adatoms and accordingly to lower the size of formed grains of thin film. We managed this problem successfully. As it is visible in fig. 4, the size of tips sharply has decreased and has made about 30-50 nm approximately at the same diameter of cells (~500 nm) and package of tips (~$4 \cdot 10^8$ cm^{-2}). The tips uniformity on height and form thus also was essentially improved.

The forming voltage decreasing will result in reduction of the size of cells and increase of their density. The AA fabrication with the size of cells more than 600 nm is connected to difficulties of high-voltage anodizing. The fabrication of AA with cells of the order 80-150 nm is a typical task. There is no problems to reduce the tip's sizes up to 20-40 nm. Thus density of tips will be $2 \cdot 10^{10}$ - $5 \cdot 10^9$ cm^{-2} and the expected diameter of tips will make up to 10 nm.

The using of heterogeneity of AA cell structure gives an opportunity to perform gated FEA with self-combined top electrode as was shown in [4]. But this process is enough combined and non-reproducible until now. Nevertheless after the appropriate perfection can appear extremely fruitful in future.

In result of carried out studies the tips with the size ~40 nm and average distance ~500 nm was fabricated. No problems is foreseen for reduction of the tip's sizes up to 20...40 nm. Average packing of realized structure was ~10^9 重$^{-2}$, the realization of the FEA with density of elements 10^{10}-10^{11} 重$^{-2}$ is under progress.

Thus, as it is visible from presented results, the described above technology allows to realize oxide-metal matrixes, which are the prototype of FEA, that under condition of realization self-aligned extracted electrode will give an opportunity to create gated FEAs with nanodimensional size of tips and interelectrode distances. Nowadays the natural technology based on AA allows to obtain ungated FEAs with tip's density that cannot be realized by traditional labour-consuming methods of microtechnology.

5. CONCLUSION

The new natural technology of FEA formation on the basis of self-organizing thin and thick films during growth is proposed and described. High density (10^8 -10^{10} 重$^{-2}$) and localization of tips (40-100 nm) without using of litography methods have been shown.

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Nanoporous Al/sub 2/O/sub 3/ membranes filled by platinum

- Pruneanu, S. Mihailescu, G. Indrea, E.
Nat. I, Cluj-Napoca, Romania

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References Cited: 3
INSPEC Accession Number: 6839332

Abstract:
Nanoporous aluminum oxide membranes have been filled by platinum. Two m were employed, one by cyclic voltammetry and other by alternative current. X diffraction studies performed on these samples have evidenced that longer electrodeposition time favour the growth of crystallites with larger size and le crystalline imperfections.

Index Terms:
alumina membranes electrodeposition crystallites porous materials nanostruct materials X-ray diffraction platinum anodised layers platinum filling nanoporo aluminum oxide membrane cyclic voltammetry alternating current X-ray diffra electrodeposition crystallite growth anodic thin film Al/sub 2/O/sub 3/

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L8	(AAO or (anodic adj alumina) or (anodic adj aluminum adj oxide))	421	L8
	DB=USPT; PLUR=YES; OP=OR		
L7	L5 and anodic$4	103	L7
L6	L5 and (AAO or (anodic adj alumina) or (anodic adj aluminum adj oxide))	0	L6
L5	L2 or L3 or L4	5067	L5
L4	((204/424 |204/425 |204/426 |204/431)!.CCLS.)	1275	L4
L3	((29/592.1 |29/593 |29/825)!.CCLS.)	1868	L3
L2	((73/1.02 |73/1.06 |73/23.2 |73/29.01 |73/29.02 |73/335.02 |73/335.03 |73/335.05 |73/31.05)!.CCLS.)	2020	L2
L1	(method and making and gas and sensor).ti.	21	L1

END OF SEARCH HISTORY